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Full Text

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TI Grinding aids for use in silicone wafer polishing slurry containing colloidal silica

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SO Jpn. Kokai Tokkyo Koho, 6 pp.

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AB

The grinding aids are of HO(PO)_a(EO)_b(PO)_cH block copolymers (EO = ethylene oxide; PO = propylene oxide, a, b, c ≥ 1) which are included for improving the stability of silica dispersion.

102b(1-2)
103-3
Mizutari et al.

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TITLE: Polishing assistant for silicon wafer, consists of a block polyether

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PATENT-FAMILY:

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INT-CL (IPC): B24B037/00, B24B057/04 , C09K003/14 , H01L021/304

ABSTRACTED-PUB-NO: JP2001110760A

BASIC-ABSTRACT:

NOVELTY - A polishing assistant for a silicon wafer consists of a block polyether.

DETAILED DESCRIPTION - The block polyether of formula (1).

HO-(EO)_d-(PO)_e-(EO)_f-H (1)

EO = oxyethylene;

PO = oxypropylene;

a, b, c = 1 or greater.

USE - The polishing assistant is suitably used for mirror surface polishing for the silicon wafer in production processes for a semiconductor.

ADVANTAGE - The polishing assistant yields an abrasive solution having superior polishing performance.

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS: POLISH ASSIST SILICON WAFER CONSIST BLOCK POLYETHER

DERWENT-CLASS: A25 A88 P61 U11

CPI-CODES: A05-H03; A05-H04; A12-E07C; A12-H;

EPI-CODES: U11-C06A1A;

ENHANCED-POLYMER-INDEXING: Polymer Index [1.1] 018 ; R00351 G1558 D01 D23 D22 D31 D42 D50 D73 D82 F47 ; R00370 G1558 D01 D11 D10 D23 D22 D31 D42 D50 D73 D83 F47 ; H0022 H0011 ; H0066 H0044 H0011 ; P0055 ; P0975*R P0964 F34 D01 D10

Polymer Index [1.2] 018 ; ND01 ; B9999 B5094 B4977 B4740 ; Q9999 Q6600 ; Q9999 Q9110 ; Q9999 Q7476 Q7330

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PAT-NO: JP02001110760A
DOCUMENT-IDENTIFIER: JP 2001110760 A
TITLE: POLISHING ASSISTANT FOR SILICON WAFER

PUBN-DATE: April 20, 2001

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ABSTRACT:

PROBLEM TO BE SOLVED: To provide a higher-performance polishing liquid for a silicon wafer so as to meet the needs in the semiconductor industry where a higher accuracy and higher quality of silicon wafer is required accompanying its development.

SOLUTION: A polishing assistant for a silicon wafer consisting of block polyether expressed by the following general formula: HO-(PO)*a*-(EO)*b*-(PO)*c*-H (in the formula, EO expresses an oxyethylene group, and PO expresses an oxypropylene group, and *a*, *b*, and *c* express numbers of one or over), and a polishing agent composition for a silicon wafer including it are provided. Using this polishing assistant will produce polishing liquid which is more excellent in polishing performance such as surface flatness, polishing speed, etc., without haze occurring on the surface of the wafer.

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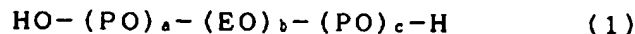
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(54) 【発明の名称】 シリコンウェハ－用研磨助剤

(57) 【要約】

【課題】 半導体産業の発展に伴い、より高精度、高品質のシリコンウェハーが求められ、それに伴いより高性



(式中、EOはオキシエチレン基を表わし、POはオキシアプロピレン基を表わし、a、b及びcは1以上の数を表わす。)で表わされるブロック型ポリエーテルからなるシリコンウェハ一用研磨助剤及びこれを含むシリコン

能のシリコンウェハー用の研磨液が求められている。

【解決手段】 本発明は、下記的一般式（１）

ウェハー用研磨剤組成物を提供する。本発明の研磨助剤を使用すると、ウェハー表面にヘイズが発生せず、表面の平坦さや研磨速度等の研磨性能のより優れた研磨液が得られる。

* * 【請求項1】 下記の一般式（1）



※る請求項1に記載のシリコンウェハー用研磨助剤。

【請求項3】 一般式(1)中のEO及びPOの重量比が、EO:PO=5:95~60:40であるブロック型ポリエーテルからなる請求項1又は2に記載のシリコンウェハ一用研磨助剤。

【請求項4】 更に、下記的一般式(2)



10★研磨工程終了時にシリコンウェハの表面にヘイズ（曇り）が生じるという問題がある。又、パウダー状シリカを研磨剤として用いるとヘイズは発生しにくい、パウダー状シリカを水溶液に均一に分散させる工程が新たに必要となり、更に、静置しておくパウダー状シリカが沈殿するために常に研磨液を攪拌している必要があった。特開平2-125413号公報、特開昭61-209909号公報、特開昭61-209910号公報等には、コロイダルシリカを用いたシリコンウェハ研磨剤及び研磨方法の改良法が提案されているが、根本的な問題の解決にはなっていないのが現状であった。

【0003】

【発明が解決しようとする課題】これらの問題を解決すべく、従来から、コロイダルシリカ等のシリコン研磨液に添加する研磨助剤が検討されてきた。例えば、特開平4-291722号公報には、コロイダルシリカ及び特定のHLB値を示す非イオン界面活性剤を含むシリコンウェハ一用研磨剤が開示されている。特開平4-291723号公報には、コロイダルシリカ及び特定のアニオン界面活性剤を含むシリコンウェハ一用研磨剤が開示されている。これらの界面活性剤は、研磨液の表面張力を調整して、研磨時に研磨されるシリコンウェハ一の研磨表面に研磨液を十分保持させるために使用されていた。しかし、半導体産業の発展の結果、より高精度、高品質のシリコンウェハ一が求められるにつれ上述のような界面活性剤ではヘイズの防止はできるが研磨表面の平坦さや研磨速度等の研磨助剤としての性能が不十分であることが指摘されはじめた。

【0004】

【課題を解決するための手段】そこで本発明者らは鋭意検討し、特定の構造を有するポリエーテルが従来の研磨助剤より優れた性能を示すことを発見し本発明を完成させた。即ち、本発明は、下記的一般式（１）

(1)

☆表わす。a、b及びcは1以上の数を表わす。a、b及びcの好ましい値の範囲は、一般式(1)で表わされるブロック型ポリエーテルの分子量及びHLB等によって相関的に変化するので一概には言えないが、a+c及びbはそれぞれ通常は2~500程度、更に好ましくは10~100程度である。一般式(1)で表わされるブロック型ポリエーテルは、エチレンオキシドをbモル重

【発明の実施の形態】一般式(1)において、EOはオキシエチレン基を表わし、POはオキシプロピレン基を☆50

* 2種以上のブロック型ポリエーテルを使用することができ、研磨液の表面張力や親水性等を微調整したい場合は、2種以上を併用して使用することが好ましい。一般式(1)で表わされるブロック型ポリエーテルの中でも、シリコンウェハを研磨する研磨液に特に好ましい表面張力を与えるものは、EO及びPOの重量比がE O : P O = 5 : 95 ~ 60 : 40であるブロック型ポリエーテルである。このようなブロック型ポリエーテルを使用した場合は、研磨液にシリコンウェハを研磨するのに特に適した表面張力を与えることができるので、ヘイズが無く、研磨表面が平滑・平坦なシリコンウェハを得ることができ、その結果高性能の半導体素子が得られる。

【0007】更に、一般式(1)で表わされるブロック型ポリエーテルは、次の一般式(2)で表わされるブロック型ポリエーテルと併用することができる。

$$O) f-H \quad (2)$$

※均粒子径は7~100 μ mのものが好ましく、アルカリ性コロイダルシリカの濃度は20~60重量%が好ましい。

【0010】本発明のシリコンウェハ用研磨助剤の添加量は特に限定されないが、水分散シリカの固形分に対して0.005〜5重量%添加することが好ましい。その他、本発明のシリコンウェハ用研磨助剤は、メタノール、エタノール、2-プロパノール等の低級アルコール；メチルセロソルブ、エチルセロソルブ、ブチルセロソルブ、メチルカルビトール、エチルカルビトール、ブチルカルビトール等の低級アルコールのアルコキシレート；高級アルコールのアルキレンオキシサイド付加物等の界面活性剤と併用することができる。本発明のシリコンウェハ用研磨助剤は、シリコンウェハのラッピング工程又はポリッシング工程の何れにも使用することができ、研磨液の表面張力を適切な値に調整することができるため、シリコンウェハ上にヘイズが無く、表面が平滑・平坦なシリコンウェハを得ることができ、その結果高性能の半導体素子が得られる。

[0011]

【実施例】以下、実施例により本発明を更に具体的に説明する。尚、以下の実施例中、％は特に記載が無い限り重量基準である。評価試験に用いた本発明の研磨助剤及び比較品は以下のとおりである。

【0012】

【表1】

表1

		ポリエーテル (A)			ポリエーテル (B)			アミノシリカ 含有率 (%)
		a+c	b	配合量 (%)	a+c	b	配合量 (%)	
本 発 明 品	1	29	26	80	32	13	20	
	2	29	28	80	32	13	40	
	3	29	26	80	31	7	20	
	4	29	26	70	36	24	30	
	5	26	15	85	53	13	15	
	6	26	15	100				
	7	53	47	70	32	13	30	
	8	53	47	80	31	7	20	
	9	29	26	55	35	24	45	
	10	29	26	60	32	13	20	20
	11	17	15	70	32	13	30	

【0013】

* * 【表2】

表2

		ポリエーテル (A)			ポリエーテル (C)			アミノシリカ 含有率 (%)
		a+c	b	配合量 (%)	d+f	e	配合量 (%)	
	12	29	26	70	9	34	30	
	13	32	13	20	33	37	60	20
	14	53	13	35	33	37	66	

【0014】※：表1及び表2において、ポリエーテル (A) 及び (B) は一般式 (1) で表わされるブロック型ポリエーテルであり、ポリエーテル (C) は一般式 (2) で表わされるブロック型ポリエーテルである。

【0015】

比較品1 アルミン酸ナトリウム

比較品2 ラウリルスルホン酸ナトリウム

比較品3 ノニルフェノールエチレンオキサイド10モル付加物

比較品4 (ブランク)

【0016】(実施例1) コロイダルシリカとしてアデライトAT-30S (商品名、旭電化工業製、平均粒子径7~10 μ m、シリカ固形分30%) を用い、これを※

※水でシリカ固形分5.0%に希釈した。これに、前記本発明の研磨助剤及び比較品をシリカ固形分に対して0.1% (比較品1は1.0%) 添加し、よく攪拌して均一化して希釈研磨液とした。これらの希釈研磨液を使用して、スライシングした4インチのシリコンウェハー100枚についてラッピング処理を行った。ラッピング処理後、100枚のシリコンウェハーについて、ウェハーの擦り傷、ウェハーの割れ、ウェハーの微小表面クラックの各不良項目に該当するものの枚数を数えた。その結果を表3に示す。

【0017】

【表3】

表3

	擦り傷 (枚)	割れ (枚)	クラック (枚)
本発明品 1	0	0	0
本発明品 2	0	0	0
本発明品 3	1	0	0
本発明品 4	1	0	0
本発明品 5	1	0	0
本発明品 6	0	0	0
本発明品 7	0	0	0
本発明品 8	1	0	0
本発明品 9	0	0	0
本発明品 10	1	0	0
本発明品 11	1	0	0
本発明品 12	1	0	0
本発明品 13	1	0	0
本発明品 14	1	0	0
比較例 1	5	5	4
比較例 2	6	4	3
比較例 3	3	5	4
比較例 4	9	10	10

【0018】(実施例2)コロイダルシリカとしてアデライトAT-30S(商品名、旭電化工業製、平均粒子径7~10 μ m、シリカ固形分30%)を用い、これを水でシリカ固形分5.0%に希釈した。これに本発明の研磨助剤及び比較品をシリカ固形分に対して0.1%(比較品1は1.0%)添加し、よく攪拌して均一化して希釈研磨液とした。この希釈研磨液を以下の条件でボ

【0019】<条件>

ポリッシングマシン：LPH-15改良機(ラップマスター製)

ポリッシング盤直径：15インチ

加工圧力：100g/cm²

加工温度：30℃

加工液供給量：10リットル/時間

加工時間：1時間

加工枚数：1枚

ポリッシャー：SUPREME RN-H(ローデル・ニッタ製)

上記条件下でファイナルポリッシングを行い、終了後、ウェハー表面を乾燥させないように注意して過酸化水素*

*-アンモニア溶液で80℃でRCA洗浄した。洗浄後、クイックダンプ洗浄を行い、スピンドライヤーで乾燥させた。乾燥後、以下の評価を行った。

【0020】評価1：ヘイズの有無

グリーンベンチ内でハロゲン平行光(超高輝度検査用照明装置U1H-1H、インテック製)のウェハー表面からの反射を目視で観察し、ヘイズの発生を観察した。

評価2：表面粗さ

研磨ウェハーの表面粗さを光学干渉式粗さ計(WYKO TOPO-3D、WYKO製、250 μ m)で平均2乗粗さを測定した。

評価3：平坦度

研磨ウェハーの表面平坦度、即ち研磨ウェハーの最大厚さと最小厚さの差(TTV)を、TTV測定装置(ADEマイクロスキャン8300、ADE製)により測定した。

40 評価4：研磨速度

研磨中のシリコンウェハーの研磨速度を研磨後のウェハーの膜厚減を7×7点測定し算出した。これらの評価結果を表4に示す。

【0021】

【表4】

表 4

	ヘイズ	表面粗さ (nm)	平坦度 (μm)	研削速度 ($\mu\text{m}/\text{分}$)
本発明品 1	無し	0.15	0.39	2.4
本発明品 2	無し	0.22	0.38	2.9
本発明品 3	無し	0.24	0.55	3.3
本発明品 4	無し	0.28	0.44	1.9
本発明品 5	無し	0.30	0.41	2.5
本発明品 6	無し	0.17	0.52	2.2
本発明品 7	無し	0.22	0.33	3.0
本発明品 8	無し	0.29	0.32	3.4
本発明品 9	無し	0.20	0.60	3.0
本発明品 10	無し	0.27	0.30	2.9
本発明品 11	無し	0.25	0.27	2.7
本発明品 12	無し	0.31	0.51	2.8
本発明品 13	無し	0.42	0.46	3.5
本発明品 14	無し	0.30	0.45	2.9
比較例 1	無し	0.88	1.42	0.8
比較例 2	無し	0.91	1.33	0.7
比較例 3	無し	0.94	1.34	0.9
比較例 4	無し	1.24	2.04	1.4

【0022】以上の実施例の各評価結果から、本発明の研磨助剤を使用した研磨液は、アニオン界面活性剤（比較品2）、非イオン界面活性剤（比較品3）を使用した研磨液に比べて、シリコンウェハの割れや擦り傷がなく、表面粗さ、平坦度、研磨速度等に優れていることが*

* 分かる.

【0023】

【発明の効果】本発明の効果は、より研磨性能の優れた研磨液を与える新規なシリコンウェハ－研磨助剤を提供したことにある。

フロントページの続き

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CLAIMS

[Claim(s)]

[Claim 1] The following general formula (1)

HO-(PO)_a-(EO)_b-(PO)_c-H (1)

(EO expresses an oxyethylene radical among a formula, PO expresses an oxypropylene radical, and a, b, and c express one or more numbers.) The polish assistant for silicon wafers which consists of a block-type polyether expressed.

[Claim 2] The polish assistant for silicon wafers according to claim 1 whose molecular weight of the block-type polyether expressed with a general formula (1) is 1,000-10,000.

[Claim 3] The polish assistant for silicon wafers according to claim 1 or 2 which the weight ratio of EO and PO in a general formula (1) becomes from the block-type polyether which is EO:PO=5:95-60:40.

[Claim 4] Furthermore, the following general formula (2)

HO-(EO)_d-(PO)_e-(EO)_f-H (2)

(-- EO expresses an oxyethylene radical among a formula, PO expresses an oxypropylene radical, and d, e, and f express one or more numbers.) -- polish assistant for silicon wafers given in claim 1 containing the block-type polyether expressed thru/or any 1 term of 3.

[Claim 5] The abrasive material constituent for silicon wafers which contains the polish assistant for silicon wafers and colloidal silica of a publication in claim 1 thru/or any 1 term of 4.

[Claim 6] The polish approach of the silicon wafer using the abrasive material constituent for silicon wafers according to claim 5.

[Claim 7] The silicon wafer ground by the polish approach of a silicon wafer according to claim 6.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the polish assistant for silicon wafers used suitable for mirror polishing of the silicon wafer in the production process of a semi-conductor.

[0002]

[Description of the Prior Art] There are a wrapping process (rough polish) which removes the cutting distorted layer and surface waviness which are produced when silicon is cut down in the shape of a wafer by cutting from a silicon ingot, and a polishing process (precision polish process) which makes the surface precision made into the purpose to lap DOSHIKONWEHA which passed through the wrapping process as mirror-polishing process of the silicon wafer for semi-conductors. Moreover, at the latter polishing process, it is classified by primary polishing processes (primary polish processes) to which a great portion of precision is made, and the final polishing process (the last polish process) to which the surface precision made into the purpose is made, and primary polishing processes may be called the primary secondary polishing processes by dividing into two depending on the case.

Conventionally, generally as an abrasive material of a silicon wafer, colloidal silica and cerium oxide have been used. However, when it grinds only using colloidal silica polish liquid or cerium oxide polish liquid, there is a problem that Hayes (cloudiness) is generated on the surface of a silicon wafer, at the time of polish process termination. Moreover, when the powder-like silica was used as an abrasive material, it was hard to generate Hayes, but if the process which homogeneity is made to distribute was newly needed for a water solution and the powder-like silica was put further, in order that a powder-like silica might precipitate, polish liquid always needed to be stirred. Although the method of improving the silicon wafer abrasive material which used colloidal silica, and the polish approach was proposed by JP,2-125413,A, JP,61-209909,A, and JP,61-209910,A, the present condition was not being solution of a fundamental problem.

[0003]

[Problem(s) to be Solved by the Invention] The polish assistant added in silicon polish liquid, such as colloidal silica, has been examined from the former that these problems should be solved. For example, the abrasive material for silicon wafers containing the nonionic surface active agent which shows colloidal silica and a specific HLB value to a publication-number No. 291722 [four to] official report is indicated. The abrasive material for silicon wafers which contains colloidal silica and a specific anionic surface active agent in a publication-number No. 291723 [four to] official report is indicated. These surfactants adjusted the surface tension of polish liquid, and they were used in order to make polish liquid hold enough on the polish front face of the silicon wafer ground at the time of polish. However, as a result of development of semiconductor industry, although prevention of Hayes was completed, with the above surfactants, it began to be pointed out that the engine performance as polish assistants, such as flat [on the front face of polish] and a polish rate, is inadequate, as the silicon wafer of high degree of accuracy and high quality was called for more.

[0004]

[Means for Solving the Problem] Then, this invention persons inquire wholeheartedly, and the polyether which has specific structure discovered that the engine performance superior to the conventional polish assistant was shown, and completed this invention. That is, this invention is the following general formula (1).



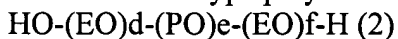
(-- EO expresses an oxyethylene radical among a formula, PO expresses an oxypropylene radical, and a, b, and c express one or more numbers.) -- it is the polish assistant for silicon wafers which consists of a block-type polyether expressed.

[0005]

[Embodiment of the Invention] In a general formula (1), EO expresses an oxyethylene radical and PO expresses an oxypropylene radical. a, b, and c express one or more numbers. Although the range of the desirable value of a, b, and c does not generally have ***** since it changes with molecular weight, HLB, etc. of a block-type polyether which are expressed with a general formula (1) interrelatively, a+c and b are usually ten to about 100 still more preferably two to about 500, respectively. The block-type polyether expressed with a general formula (1) can be obtained by carrying out a+c mol addition of the propylene oxide to the polyethylene glycol which was made to carry out the b mol polymerization of the ethyleneoxide, and was obtained. Under the present circumstances, since the physical properties of the block-type polyether obtained, for example, surface tension, a hydrophilic property, etc. are controllable by the molecular weight of the polyethylene glycol used as the base, the amount of propylene oxide made to add, it is possible to use it, choosing the block-type polyether suitable for the silicon wafer ground or the approach of grinding.

[0006] Moreover, although especially the molecular weight of the block-type polyether expressed with a general formula (1) is not limited, if the effectiveness of adjusting the surface tension of polish liquid if too not much small etc. becomes inadequate and is too large not much, since the viscosity of the block-type polyether expressed with a general formula (1) will rise too much, 1,000-10,000 are desirable. Two or more sorts of block-type polyethers from which the value of a, b, or c differs can be used as well as using one sort independently, and, as for the block-type polyether expressed with a general formula (1), it is desirable to use together and use two or more sorts to tune surface tension, a hydrophilic property, etc. of polish liquid finely. Especially the thing that gives desirable surface tension to the polish liquid which grinds a silicon wafer also in the block-type polyether expressed with a general formula (1) is a block-type polyether whose weight ratio of EO and PO is EO:PO=5:95-60:40. Since the surface tension which was suitable for polish liquid especially grinding a silicon wafer can be given when such a block-type polyether is used, there is not Hayes, a silicon wafer smooth [a polish front face] and flat can be obtained, and, as a result, the semiconductor device of high performance is obtained.

[0007] Furthermore, the block-type polyether expressed with a general formula (1) can be used together with the block-type polyether expressed with the following general formula (2).



In a general formula (2), d, e, and f express one or more numbers. Although the range of the desirable value of d, e, and f does not generally have ***** since it changes with molecular weight, HLB, etc. of a block-type polyether which are expressed with a general formula (2) interrelatively, d+f and e are usually ten to about 100 still more preferably two to about 500, respectively. The block-type polyether expressed with a general formula (2) can be obtained by carrying out d+f mol addition of the ethyleneoxide to the polypropylene glycol which was made to carry out the e mol polymerization of the propylene oxide, and was obtained.

[0008] Moreover, when glycol system solvents, such as propylene glycol, ethylene glycol, and butanediol, are added to it, since the viscosity of polish liquid is lowered to the block-type polyether expressed with a general formula (1) and the dispersibility of moisture powder silicas, such as colloidal silica, is raised to it, it is still more desirable to it. When using together a block-type polyether and a glycol system solvent, it is desirable to make a glycol system solvent into 1 - 40 % of the weight to the constituent whole quantity 60 to 99% of the weight to the whole quantity of the constituent which consists a block-type polyether of a block-type polyether and a glycol system solvent.

[0009] The polish assistant for silicon wafers of above-mentioned this invention is usually added and used for the moisture powder silica which is an abrasive material. As a moisture powder silica, although a moisture powder powder silica, colloidal silica, etc. are mentioned for example, the alkaline colloidal silica adjusted to pH 8-12 especially is desirable. Also in alkaline colloidal silica, the 7-100-micrometer thing of mean particle diameter is desirable, and the concentration of alkaline colloidal silica has 20 - 60 desirable % of the weight.

[0010] Although especially the addition of the polish assistant for silicon wafers of this invention is not limited, it is desirable to add 0.005 to 5% of the weight to the solid content of a moisture powder silica. In addition, the silicon wafer polish assistant of this invention can be used together with surfactants, such as an alkylene oxide addition product of alkoxylate; higher alcohol of lower alcohol, such as lower alcohol; methyls cellosolve, such as a methanol, ethanol, and 2-propanol, ethylcellosolve, butyl cellosolve, methyl carbitol, ethyl carbitol, and butyl carbitol. Since the silicon wafer polish assistant of this invention can be used for either the wrapping process of a silicon wafer, or a polishing process and the surface tension of polish liquid can be adjusted to a suitable value, Hayes is not on a silicon wafer, a silicon wafer smooth [a front face] and flat can be obtained, and, as a result, the semiconductor device of high performance is obtained.

[0011]

[Example] Hereafter, an example explains this invention still more concretely. In addition, among the following examples, especially % is weight criteria, as long as it is unstated. The polish assistant and comparison article of this invention which were used for the evaluation trial are as follows.

[0012]

[Table 1]

表 1

		ポリエーテル (A)			ポリエーテル (B)			7°ピリッ グリコール (%)
		a + c	b	配合量 (%)	a + c	b	配合量 (%)	
本 発 明 の 品	1	29	26	80	32	13	20	
	2	29	26	60	32	13	40	
	3	29	26	80	31	7	20	
	4	29	26	70	35	24	30	
	5	26	15	85	53	13	15	
	6	26	15	100				
	7	53	47	70	32	13	30	
	8	53	47	80	31	7	20	
	9	29	26	55	35	24	45	
	10	29	26	60	32	13	20	20
	11	17	15	70	32	13	30	

[0013]

[Table 2]

表 2

		ポリエーテル (A)			ポリエーテル (C)			7°ピリッ グリコール (%)
		a + c	b	配合量 (%)	d + f	e	配合量 (%)	
	12	29	26	70	9	34	30	
	13	32	13	20	33	37	60	20
	14	53	13	35	33	37	65	

[0014] * : in Table 1 and 2, a polyether (A) and (B) are block-type polyethers expressed with a general formula (1), and a polyether (C) is a block-type polyether expressed with a general formula (2).

[0015]

Comparison article 1 Sodium-aluminate comparison article 2 Lauryl sulfonic-acid sodium comparison article 3 Ten mol addition product comparison article 4 of nonyl phenol ethyleneoxides (blank)
 [0016] (Example 1) This was diluted to 5.0% of silica solid content with water, using ADERAITO AT-30S (a trade name, the Asahi Denka Kogyo make, mean particle diameter of 7-10 micrometers, 30% of silica solid content) as colloidal silica. Addition of the polish assistant and comparison article of said this invention was carried out 0.1% (the comparison article 1 is 1.0%) to silica solid content, and to this, it stirred well, equalized to it, and considered as dilution polish liquid at it. These dilution polish liquid was used and wrapping processing was performed about 100 4 inches silicon wafers which carried out slicing. After wrapping processing, about the silicon wafer of 100 sheets, although it corresponded to each defect item of the abrasion of a wafer, the crack of a wafer, and the minute surface crack of a wafer, number of sheets was counted. The result is shown in Table 3.

[0017]

[Table 3]

表 3

	擦り傷 (枚)	割れ (枚)	クラック (枚)
本発明品 1	0	0	0
本発明品 2	0	0	0
本発明品 3	1	0	0
本発明品 4	1	0	0
本発明品 5	1	0	0
本発明品 6	0	0	0
本発明品 7	0	0	0
本発明品 8	1	0	0
本発明品 9	0	0	0
本発明品 10	1	0	0
本発明品 11	1	0	0
本発明品 12	1	0	0
本発明品 13	1	0	0
本発明品 14	1	0	0
比較例 1	5	5	4
比較例 2	6	4	3
比較例 3	3	5	4
比較例 4	9	10	10

[0018] (Example 2) This was diluted to 5.0% of silica solid content with water, using ADERAITO AT-30S (a trade name, the Asahi Denka Kogyo make, mean particle diameter of 7-10 micrometers, 30% of silica solid content) as colloidal silica. Addition of the polish assistant and comparison article of this invention was carried out 0.1% (the comparison article 1 is 1.0%) to silica solid content at this, and it stirred well, equalized and considered as dilution polish liquid. This dilution polish liquid was slushed into the polishing machine on condition that the following, and the primary polishing silicon wafer which passed through primary polishing processes was ground.

[0019] <Condition> polishing machine: LPH-15 amelioration machine (product made from a lap master)

polishing board diameter: -- 15 inch processing pressure force: -- 100g[/cm] 2 working temperature: -- 30-degree-C working liquid amount-of-supply: -- 10l. [/] hour floor-to-floor-time: -- 1-hour processing number-of-sheets: -- one-sheet polisher: -- SUPREME RN-H (the Rodel Nitta make)

Final polishing was performed under the above-mentioned conditions, after termination, it warned against drying a wafer front face, and RCA washing was carried out at 80 degrees C with hydrogen-peroxide-ammonia liquor. Quick discharge washing was performed after washing and it was made to dry with a spin dryer. The following evaluations were performed after desiccation.

[0020] Evaluation 1: The reflection from the wafer front face of halogen parallel light (super-high brightness checking lighting system U1 H-1 H, Intec make) was visually observed within the existence Green bench of Hayes, and generating of Hayes was observed.

Evaluation 2: Granularity was measured for the surface roughness of a surface roughness polish wafer the 2nd [an average of] power with the optical interference type granularity plan (WYKOTOPO-3D, the product made from WYKO, 250 micrometers).

Evaluation 3: The difference (TTV) of the surface display flatness of a display flatness polish wafer, i.e., the maximum thickness of a polish wafer, and the minimum thickness was measured with the TTV measuring device (the ADE micro scan 8300, product made from ADE).

Evaluation 4: 7x7 decreases of thickness of the wafer after grinding the polish rate of the silicon wafer under polish rate polish were measured, and were computed. These evaluation results are shown in Table 4.

[0021]

[Table 4]

表 4

	ヘイズ	表面粗さ (nm)	平坦度 (μm)	研磨速度 ($\mu\text{m}/\text{分}$)
本発明品 1	無し	0.15	0.39	2.4
本発明品 2	無し	0.22	0.38	2.9
本発明品 3	無し	0.24	0.55	3.3
本発明品 4	無し	0.28	0.44	1.9
本発明品 5	無し	0.30	0.41	2.5
本発明品 6	無し	0.17	0.52	2.2
本発明品 7	無し	0.22	0.33	3.0
本発明品 8	無し	0.29	0.32	3.4
本発明品 9	無し	0.20	0.60	3.0
本発明品 10	無し	0.27	0.30	2.9
本発明品 11	無し	0.25	0.27	2.7
本発明品 12	無し	0.31	0.51	2.8
本発明品 13	無し	0.42	0.46	3.5
本発明品 14	無し	0.30	0.45	2.9
比較例 1	無し	0.88	1.42	0.8
比較例 2	無し	0.91	1.33	0.7
比較例 3	無し	0.94	1.34	0.9
比較例 4	無し	1.24	2.04	1.4

[0022] Each evaluation result of the above example shows the polish liquid which used the polish assistant of this invention not having the crack or abrasion of a silicon wafer compared with the polish liquid which used the anionic surface active agent (comparison article 2) and the nonionic surface active agent (comparison article 3), and excelling in surface roughness, display flatness, a polish rate, etc.

[0023]

[Effect of the Invention] The effectiveness of this invention is to have offered the new silicon wafer polish assistant which gives the polish liquid which was more excellent in the polish engine performance.

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TECHNICAL FIELD

[Field of the Invention] This invention relates to the polish assistant for silicon wafers used suitable for mirror polishing of the silicon wafer in the production process of a semi-conductor.

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PRIOR ART

[Description of the Prior Art] There are a wrapping process (rough polish) which removes the cutting distorted layer and surface waviness which are produced when silicon is cut down in the shape of a wafer by cutting from a silicon ingot, and a polishing process (precision polish process) which makes the surface precision made into the purpose to lap DOSHIKONWEHA which passed through the wrapping process as mirror-polishing process of the silicon wafer for semi-conductors. Moreover, at the latter polishing process, it is classified by primary polishing processes (primary polish processes) to which a great portion of precision is made, and the final polishing process (the last polish process) to which the surface precision made into the purpose is made, and primary polishing processes may be called the primary secondary polishing processes by dividing into two depending on the case. Conventionally, generally as an abrasive material of a silicon wafer, colloidal silica and cerium oxide have been used. However, when it grinds only using colloidal silica polish liquid or cerium oxide polish liquid, there is a problem that Hayes (cloudiness) is generated on the surface of a silicon wafer, at the time of polish process termination. Moreover, when the powder-like silica was used as an abrasive material, it was hard to generate Hayes, but if the process which homogeneity is made to distribute was newly needed for a water solution and the powder-like silica was put further, in order that a powder-like silica might precipitate, polish liquid always needed to be stirred. Although the method of improving the silicon wafer abrasive material which used colloidal silica, and the polish approach was proposed by JP,2-125413,A, JP,61-209909,A, and JP,61-209910,A, the present condition was not being solution of a fundamental problem.

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EFFECT OF THE INVENTION

[Effect of the Invention] The effectiveness of this invention is to have offered the new silicon wafer polish assistant which gives the polish liquid which was more excellent in the polish engine performance.

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] The polish assistant added in silicon polish liquid, such as colloidal silica, has been examined from the former that these problems should be solved. For example, the abrasive material for silicon wafers containing the nonionic surface active agent which shows colloidal silica and a specific HLB value to a publication-number No. 291722 [four to] official report is indicated. The abrasive material for silicon wafers which contains colloidal silica and a specific anionic surface active agent in a publication-number No. 291723 [four to] official report is indicated. These surfactants adjusted the surface tension of polish liquid, and they were used in order to make polish liquid hold enough on the polish front face of the silicon wafer ground at the time of polish. However, as a result of development of semiconductor industry, although prevention of Hayes was completed, with the above surfactants, it began to be pointed out that the engine performance as polish assistants, such as flat [on the front face of polish] and a polish rate, is inadequate, as the silicon wafer of high degree of accuracy and high quality was called for more.

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MEANS

[Means for Solving the Problem] Then, this invention persons inquire wholeheartedly, and the polyether which has specific structure discovered that the engine performance superior to the conventional polish assistant was shown, and completed this invention. That is, this invention is the following general formula (1).

HO-(PO)*a*-(EO)*b*-(PO)*c*-H (1)

(-- EO expresses an oxyethylene radical among a formula, PO expresses an oxypropylene radical, and *a*, *b*, and *c* express one or more numbers.) -- it is the polish assistant for silicon wafers which consists of a block-type polyether expressed.

[0005]

[Embodiment of the Invention] In a general formula (1), EO expresses an oxyethylene radical and PO expresses an oxypropylene radical. *a*, *b*, and *c* express one or more numbers. Although the range of the desirable value of *a*, *b*, and *c* does not generally have ***** since it changes with molecular weight, HLB, etc. of a block-type polyether which are expressed with a general formula (1) interrelatively, *a*+*c* and *b* are usually ten to about 100 still more preferably two to about 500, respectively. The block-type polyether expressed with a general formula (1) can be obtained by carrying out *a*+*c* mol addition of the propylene oxide to the polyethylene glycol which was made to carry out the *b* mol polymerization of the ethyleneoxide, and was obtained. Under the present circumstances, since the physical properties of the block-type polyether obtained, for example, surface tension, a hydrophilic property, etc. are controllable by the molecular weight of the polyethylene glycol used as the base, the amount of propylene oxide made to add, it is possible to use it, choosing the block-type polyether suitable for the silicon wafer ground or the approach of grinding.

[0006] Moreover, although especially the molecular weight of the block-type polyether expressed with a general formula (1) is not limited, if the effectiveness of adjusting the surface tension of polish liquid if too not much small etc. becomes inadequate and is too large not much, since the viscosity of the block-type polyether expressed with a general formula (1) will rise too much, 1,000-10,000 are desirable. Two or more sorts of block-type polyethers from which the value of *a*, *b*, or *c* differs can be used as well as using one sort independently, and, as for the block-type polyether expressed with a general formula (1), it is desirable to use together and use two or more sorts to tune surface tension, a hydrophilic property, etc. of polish liquid finely. Especially the thing that gives desirable surface tension to the polish liquid which grinds a silicon wafer also in the block-type polyether expressed with a general formula (1) is a block-type polyether whose weight ratio of EO and PO is EO:PO=5:95-60:40. Since the surface tension which was suitable for polish liquid especially grinding a silicon wafer can be given when such a block-type polyether is used, there is not Hayes, a silicon wafer smooth [a polish front face] and flat can be obtained, and, as a result, the semiconductor device of high performance is obtained.

[0007] Furthermore, the block-type polyether expressed with a general formula (1) can be used together with the block-type polyether expressed with the following general formula (2).

HO-(EO)*d*-(PO)*e*-(EO)*f*-H (2)

In a general formula (2), *d*, *e*, and *f* express one or more numbers. Although the range of the desirable

value of d, e, and f does not generally have ***** since it changes with molecular weight, HLB, etc. of a block-type polyether which are expressed with a general formula (2) interrelatively, d+f and e are usually ten to about 100 still more preferably two to about 500, respectively. The block-type polyether expressed with a general formula (2) can be obtained by carrying out d+f mol addition of the ethyleneoxide to the polypropylene glycol which was made to carry out the e mol polymerization of the propylene oxide, and was obtained.

[0008] Moreover, when glycol system solvents, such as propylene glycol, ethylene glycol, and butanediol, are added to it, since the viscosity of polish liquid is lowered to the block-type polyether expressed with a general formula (1) and the dispersibility of moisture powder silicas, such as colloidal silica, is raised to it, it is still more desirable to it. When using together a block-type polyether and a glycol system solvent, it is desirable to make a glycol system solvent into 1 - 40 % of the weight to the constituent whole quantity 60 to 99% of the weight to the whole quantity of the constituent which consists a block-type polyether of a block-type polyether and a glycol system solvent.

[0009] The polish assistant for silicon wafers of above-mentioned this invention is usually added and used for the moisture powder silica which is an abrasive material. As a moisture powder silica, although a moisture powder powder silica, colloidal silica, etc. are mentioned for example, the alkaline colloidal silica adjusted to pH 8-12 especially is desirable. Also in alkaline colloidal silica, the 7-100-micrometer thing of mean particle diameter is desirable, and the concentration of alkaline colloidal silica has 20 - 60 desirable % of the weight.

[0010] Although especially the addition of the polish assistant for silicon wafers of this invention is not limited, it is desirable to add 0.005 to 5% of the weight to the solid content of a moisture powder silica. In addition, the silicon wafer polish assistant of this invention can be used together with surfactants, such as an alkylene oxide addition product of alkoxylate; higher alcohol of lower alcohol, such as lower alcohol; methyls cellosolve, such as a methanol, ethanol, and 2-propanol, ethylcellosolve, butyl cellosolve, methyl carbitol, ethyl carbitol, and butyl carbitol. Since the silicon wafer polish assistant of this invention can be used for either the wrapping process of a silicon wafer, or a polishing process and the surface tension of polish liquid can be adjusted to a suitable value, Hayes is not on a silicon wafer, a silicon wafer smooth [a front face] and flat can be obtained, and, as a result, the semiconductor device of high performance is obtained.

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EXAMPLE

[Example] Hereafter, an example explains this invention still more concretely. In addition, among the following examples, especially % is weight criteria, as long as it is unstated. The polish assistant and comparison article of this invention which were used for the evaluation trial are as follows.

[0012]

[Table 1]

表1

		ポリエーテル (A)			ポリエーテル (B)			アミノシリコーン (%)
		a + c	b	配合量 (%)	a + c	b	配合量 (%)	
本発明品	1	29	26	80	32	13	20	
	2	29	28	60	32	13	40	
	3	29	26	80	31	7	20	
	4	29	26	70	35	24	30	
	5	26	15	85	53	13	15	
	6	26	15	100				
	7	53	47	70	32	13	30	
	8	53	47	80	31	7	20	
	9	29	26	55	35	24	45	
	10	29	26	60	32	13	20	20
	11	17	15	70	32	13	30	

[0013]

[Table 2]

表2

		ポリエーテル (A)			ポリエーテル (C)			アミノシリコーン (%)
		a + c	b	配合量 (%)	d + f	e	配合量 (%)	
	12	29	26	70	9	34	30	
	13	32	13	20	33	37	60	20
	14	53	13	35	33	37	65	

[0014] * : in Table 1 and 2, a polyether (A) and (B) are block-type polyethers expressed with a general formula (1), and a polyether (C) is a block-type polyether expressed with a general formula (2).

[0015]

Comparison article 1 Sodium-aluminate comparison article 2 Lauryl sulfonic-acid sodium comparison article 3 Ten mol addition product comparison article 4 of nonyl phenol ethyleneoxides (blank)

[0016] (Example 1) This was diluted to 5.0% of silica solid content with water, using ADERAITO AT-30S (a trade name, the Asahi Denka Kogyo make, mean particle diameter of 7-10 micrometers, 30% of

silica solid content) as colloidal silica. Addition of the polish assistant and comparison article of said this invention was carried out 0.1% (the comparison article 1 is 1.0%) to silica solid content, and to this, it stirred well, equalized to it, and considered as dilution polish liquid at it. These dilution polish liquid was used and wrapping processing was performed about 100 4 inches silicon wafers which carried out slicing. After wrapping processing, about the silicon wafer of 100 sheets, although it corresponded to each defect item of the abrasion of a wafer, the crack of a wafer, and the minute surface crack of a wafer, number of sheets was counted. The result is shown in Table 3.

[0017]

[Table 3]

表 3

	擦り傷 (枚)	割れ (枚)	クラック (枚)
本発明品 1	0	0	0
本発明品 2	0	0	0
本発明品 3	1	0	0
本発明品 4	1	0	0
本発明品 5	1	0	0
本発明品 6	0	0	0
本発明品 7	0	0	0
本発明品 8	1	0	0
本発明品 9	0	0	0
本発明品 10	1	0	0
本発明品 11	1	0	0
本発明品 12	1	0	0
本発明品 13	1	0	0
本発明品 14	1	0	0
比較例 1	5	5	4
比較例 2	6	4	3
比較例 3	3	5	4
比較例 4	9	10	10

[0018] (Example 2) This was diluted to 5.0% of silica solid content with water, using ADERAITO AT-30S (a trade name, the Asahi Denka Kogyo make, mean particle diameter of 7-10 micrometers, 30% of silica solid content) as colloidal silica. Addition of the polish assistant and comparison article of this invention was carried out 0.1% (the comparison article 1 is 1.0%) to silica solid content at this, and it stirred well, equalized and considered as dilution polish liquid. This dilution polish liquid was slushed into the polishing machine on condition that the following, and the primary polishing silicon wafer which passed through primary polishing processes was ground.

[0019] <Condition> polishing machine: LPH-15 amelioration machine (product made from a lap master)

polishing board diameter: -- 15 inch processing pressure force: -- 100g[/cm] 2 working temperature: -- 30-degree-C working liquid amount-of-supply: -- 10l. [/] hour floor-to-floor-time: -- 1-hour processing number-of-sheets: -- one-sheet polisher: -- SUPREME RN-H (the Rodel Nitta make)

Final polishing was performed under the above-mentioned conditions, after termination, it warned against drying a wafer front face, and RCA washing was carried out at 80 degrees C with hydrogen-peroxide-ammonia liquor. Quick discharge washing was performed after washing and it was made to dry with a spin dryer. The following evaluations were performed after desiccation.

[0020] Evaluation 1: The reflection from the wafer front face of halogen parallel light (super-high brightness checking lighting system U1 H-1 H, Intec make) was visually observed within the existence Green bench of Hayes, and generating of Hayes was observed.

Evaluation 2: Granularity was measured for the surface roughness of a surface roughness polish wafer the 2nd [an average of] power with the optical interference type granularity plan (WYKOTOPO-3D, the product made from WYKO, 250 micrometers).

Evaluation 3: The difference (TTV) of the surface display flatness of a display flatness polish wafer, i.e., the maximum thickness of a polish wafer, and the minimum thickness was measured with the TTV measuring device (the ADE micro scan 8300, product made from ADE).

Evaluation 4: 7x7 decreases of thickness of the wafer after grinding the polish rate of the silicon wafer under polish rate polish were measured, and were computed. These evaluation results are shown in Table 4.

[0021]

[Table 4]

表 4

	ヘイズ	表面粗さ (nm)	平坦度 (μm)	研磨速度 ($\mu\text{m}/\text{分}$)
本発明品 1	無し	0.15	0.39	2.4
本発明品 2	無し	0.22	0.38	2.9
本発明品 3	無し	0.24	0.55	3.3
本発明品 4	無し	0.28	0.44	1.9
本発明品 5	無し	0.30	0.41	2.5
本発明品 6	無し	0.17	0.52	2.2
本発明品 7	無し	0.22	0.33	3.0
本発明品 8	無し	0.29	0.32	3.4
本発明品 9	無し	0.20	0.60	3.0
本発明品 10	無し	0.27	0.30	2.9
本発明品 11	無し	0.25	0.27	2.7
本発明品 12	無し	0.31	0.51	2.8
本発明品 13	無し	0.42	0.46	3.5
本発明品 14	無し	0.30	0.45	2.9
比較例 1	無し	0.88	1.42	0.8
比較例 2	無し	0.91	1.33	0.7
比較例 3	無し	0.94	1.34	0.9
比較例 4	無し	1.24	2.04	1.4

[0022] Each evaluation result of the above example shows the polish liquid which used the polish assistant of this invention not having the crack or abrasion of a silicon wafer compared with the polish liquid which used the anionic surface active agent (comparison article 2) and the nonionic surface active agent (comparison article 3), and excelling in surface roughness, display flatness, a polish rate, etc.

[Translation done.]